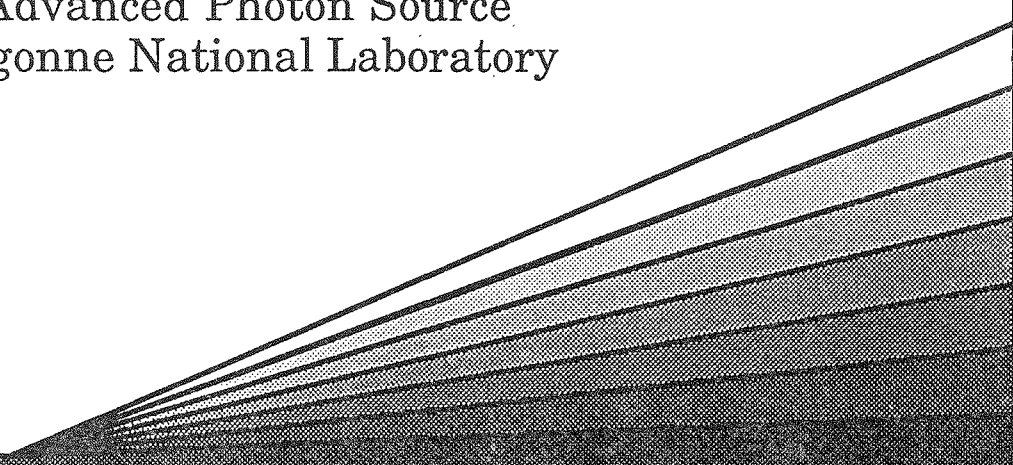


Preliminary Cleaning Tests on Candidate Materials for APS Beamline and Front End UHV Components

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Introduction

Comparative cleaning tests have been done on four candidate materials for use in APS beamline and front-end vacuum components. These materials are 304 SS, 304L SS, OFHC copper, and Glidcop* (Cu - Al₂O₃). Samples of each material were prepared and cleaned using two different methods. After cleaning, the sample surfaces were analyzed using ESCA (Electron Spectroscopy for Chemical Analysis). Uncleaned samples were used as a reference. The cleaning methods and surface analysis results are further discussed.

Cleaning Methods

The first cleaning method consisted of conventional chemical cleaning in trichlorethane, acetone, and ethyl alcohol. Samples were ultrasonically cleaned in each chemical for 15 minutes at room temperature.

The second cleaning method is a new technique not previously explored for UHV applications[1]. A remote variation of this technique using CO₂ snow as a high velocity jet to clean vacuum surfaces has been known [2]. The new technique consists of supercritical fluid cleaning using CO₂. Samples were taken to the Liquid Carbonic Supercritical Processing Facility in Allentown, Pennsylvania, where two cleaning runs at different pressures were completed.

Cleaning in chemical solvents consists essentially of dissolving the contaminants and flushing them from the surface using turbulence. Supercritical cleaning relates to the properties of gas liquification. If a pure gas is compressed below a "critical temperature," liquification occurs. At temperatures above this "critical temperature," no liquification is possible regardless of the pressure applied. Fig. 1 is a phase diagram of a typical supercritical fluid. For CO₂, the critical temperature is 31° C, and the critical pressure is 1073 psi.

As the pressure is increased, the gas density is increased to near liquid densities where the supercritical fluid displays good solubilizing properties. The

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cleaning effectiveness of supercritical CO₂ is dependent on the temperature, pressure, flow rate, turbulence, and time the materials to be cleaned remain in the media.

Supercritical cleaning in CO₂ has many attractive characteristics. CO₂ is readily available, inexpensive, non-flammable, non-toxic, and non-halogenated. Most important, it is a naturally occurring, environmentally safe gas. Its use for cleaning would eliminate the expense and documentation required for disposal of toxic spent cleaning chemicals.

Fig. 2 is a schematic of a supercritical cleaning system [3]. The material to be cleaned is placed in the cleaning vessel, which is then sealed and purged with low pressure CO₂ gas. Gas recycled from the separator stage is sent through high pressure pumps to a heat exchanger to reach the operating pressure and temperature within the supercritical fluid region. The supercritical fluid then passes through the cleaning vessel where various mixing or agitating means promote intimate contact with the material to be cleaned. After passing through the cleaning vessel, the CO₂ pressure and temperature are adjusted to allow separation of organic materials from the CO₂ (now a gas) in the separator stage. The cycle is then repeated in a closed loop, and the CO₂ is recycled through the system.

The single pass mode was used to clean the samples for this test. In this mode, the CO₂ is converted to supercritical conditions as in the closed-loop mode, but, after passing through the cleaning vessel, it is returned to the gas phase and exhausted to the atmosphere.

Two runs were completed with the supercritical fluid process. The parameters of the first run were 3500 psi cleaning vessel pressure at 45° C with a velocity through the vessel of 1.4 cm/min. The parameters of the second run were 2000 psi cleaning vessel pressure at 45° C with a velocity through the vessel of 1.6 cm/min.

Surface Analysis

Analysis of uncleaned sample surfaces proved the SS samples much more contaminated with hydrocarbons than the copper or Glidcop samples. The uncleaned SS surfaces showed only C, O, and Na with no traces of iron or chromium. Uncleaned copper and Glidcop surfaces contained C, O, Si, and reasonable traces of copper.

The chemically cleaned samples produced the "cleanest" surfaces with

approximately 1% C on the SS surfaces and < 1% C remaining on the copper and Glidcop surfaces.

Figs. 3-6 are sample ESCA profiles for each of the materials tested. Several plots such as this were done for each sample. In order to compress the data to an easier to understand format, all ESCA data were converted to atomic elemental percentages as shown in Fig. 7.

The surfaces of the supercritically cleaned SS samples contained a C residue of approximately 5.5 to 7% in comparison to 1% on the chemically cleaned samples. There were also traces of Ca and Na that were removed with chemical cleaning but not removed (1 -5%) with supercritical cleaning.

In the case of the copper and Glidcop, the C residue for the supercritically cleaned samples was 2 - 4% in comparison to < 1% for the chemically cleaned samples. Traces of Si were detected in the supercritically cleaned samples but were not detected in the chemically cleaned samples.

Conclusions

The results of both runs using the supercritical fluid process were very similar with perhaps a slight superiority in the second run. This seems somewhat contradictory to what would be expected since the pressure in the first run was higher, which means that the CO₂ density was higher. The velocity through the vessel was slightly greater in the second run, which could have created more turbulence and slightly better cleaning. The turbulence was minimal in both runs due to the low solvent velocities.

Considering the low solvent velocities, the cleaning results are impressive. Plans are underway for an additional supercritical cleaning run with higher flow rates and turbulence supplied by either an impeller or ultrasonics in an attempt to equal or exceed the chemical cleaning results. Additionally, thermal and photon induced desorption measurements will be conducted on the samples in a synchrotron facility.

Acknowledgments

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The interest and efforts of Mr. Raymond Robey and Mr. Richard Wildasin from the Supercritical Processing Group of Liquid Carbonic are greatly

appreciated for supplying the equipment and expertise to do the supercritical cleaning.

Appreciation is also expressed to Dr. John Gavrilovic from McCrone Associates who carried out the ESCA analysis under contract and added greatly to the understanding of the results.

References

1. Introduction to Supercritical Cleaning, Report by Liquid Carbonic, Supercritical Processing Group, Allentown, PA.
2. L. Layden and D. Wadlow, "High Velocity CO₂ Snow for Cleaning Vacuum System Surfaces," J. Vac. Sci. Tech., A8(5), Sept/Oct. 1990.
3. Qualifications Manual for Supercritical Cleaning, Report by Liquid Carbonic, Supercritical Processing Group, Allentown, PA.

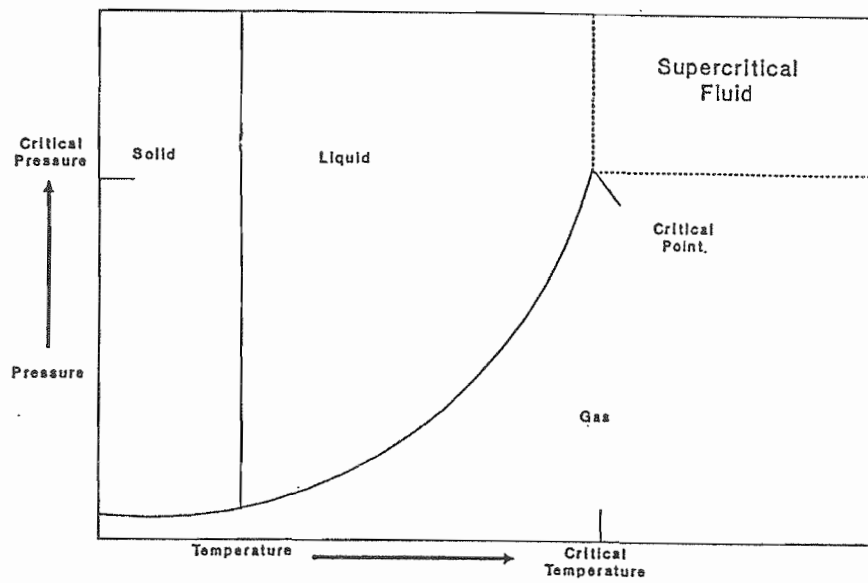


Fig. 1. Phase Diagram

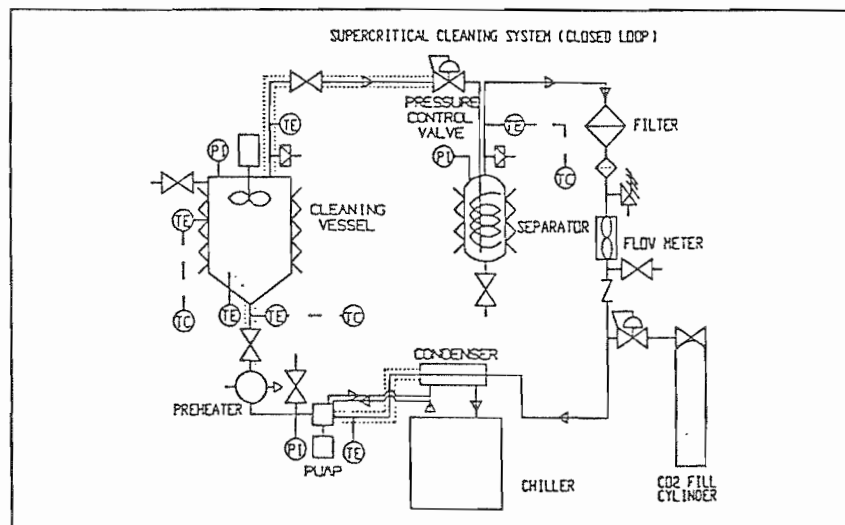


Fig. 2. Supercritical Cleaning System.

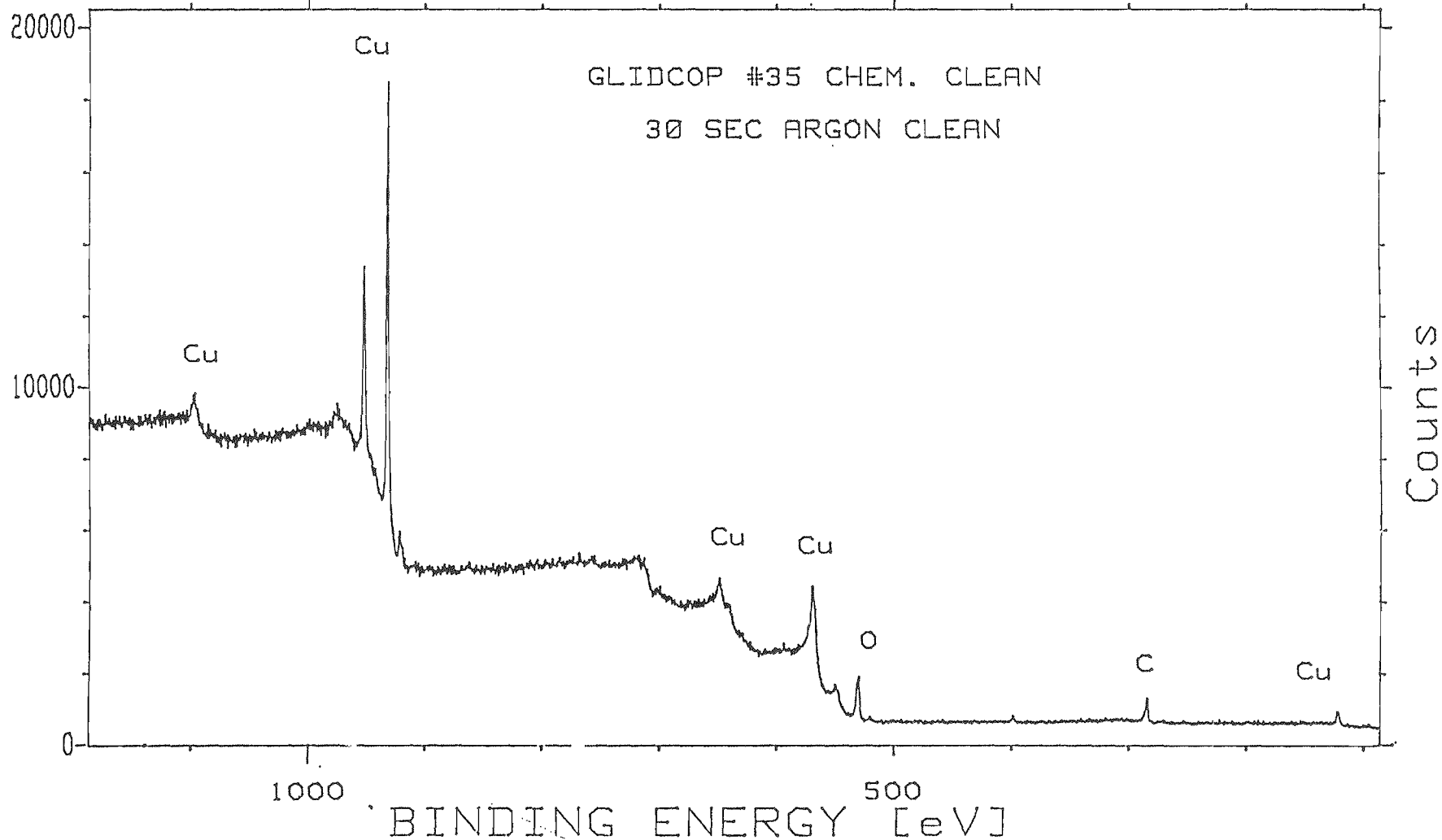


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ESCA



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Source Power 300 W Step 4

ARGON41_SP

RESO= 1.0eV

Al Anode

13:52:49

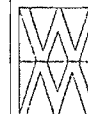
7 Apr 1992

Fig. 3. Glidcop Sample #35



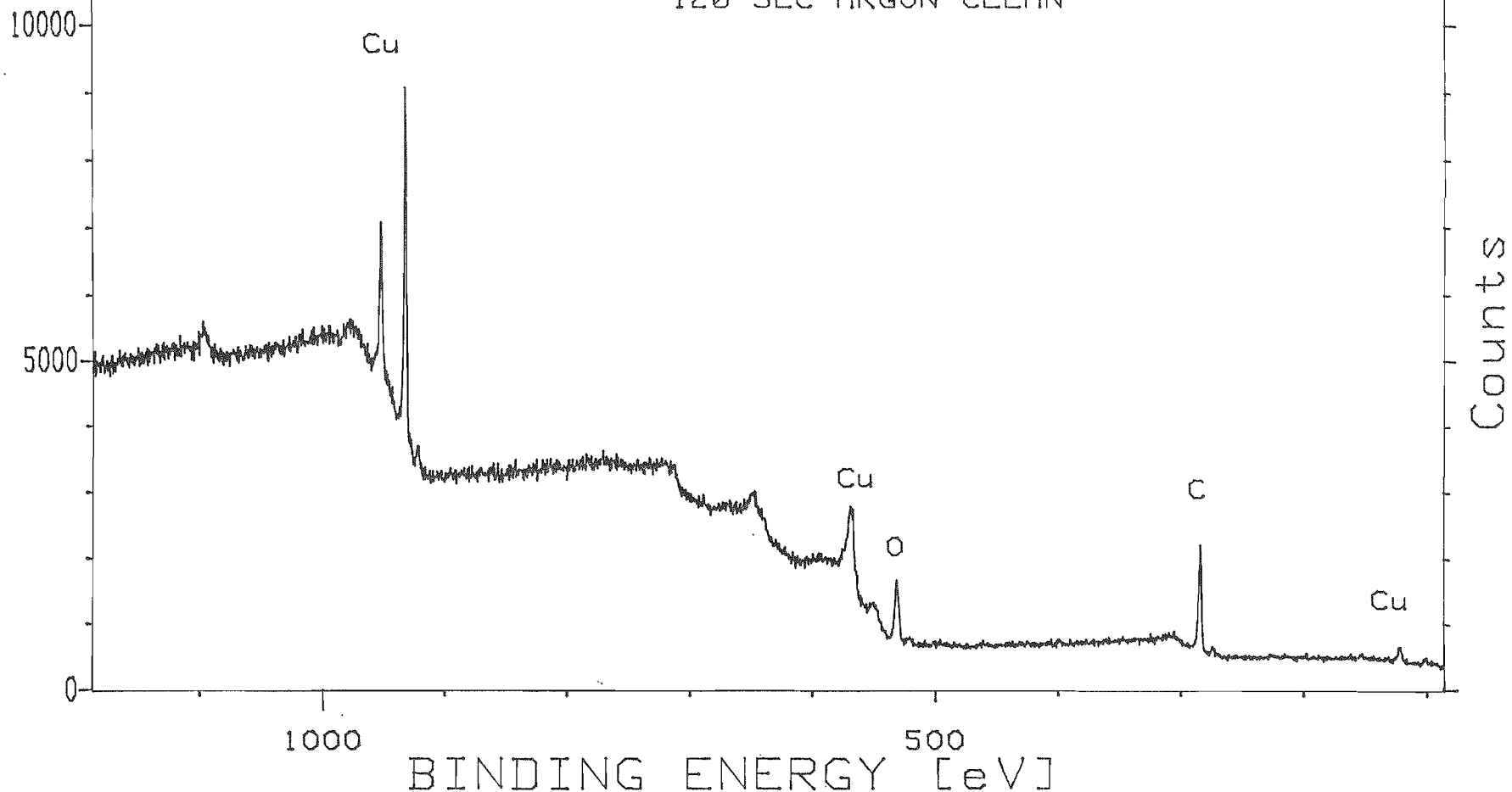
SURFACES
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ESCA



McCRONE
ASSOCIATES, INC.

OFHC #1 SINGLE PASS
120 SEC ARGON CLEAN



Source Power 300 W Step .4

RESO= 1.0eV Al Anode

ARGON19_SP

.15:31:45 6 Apr 1992

Fig. 4. OFHC Sample #1

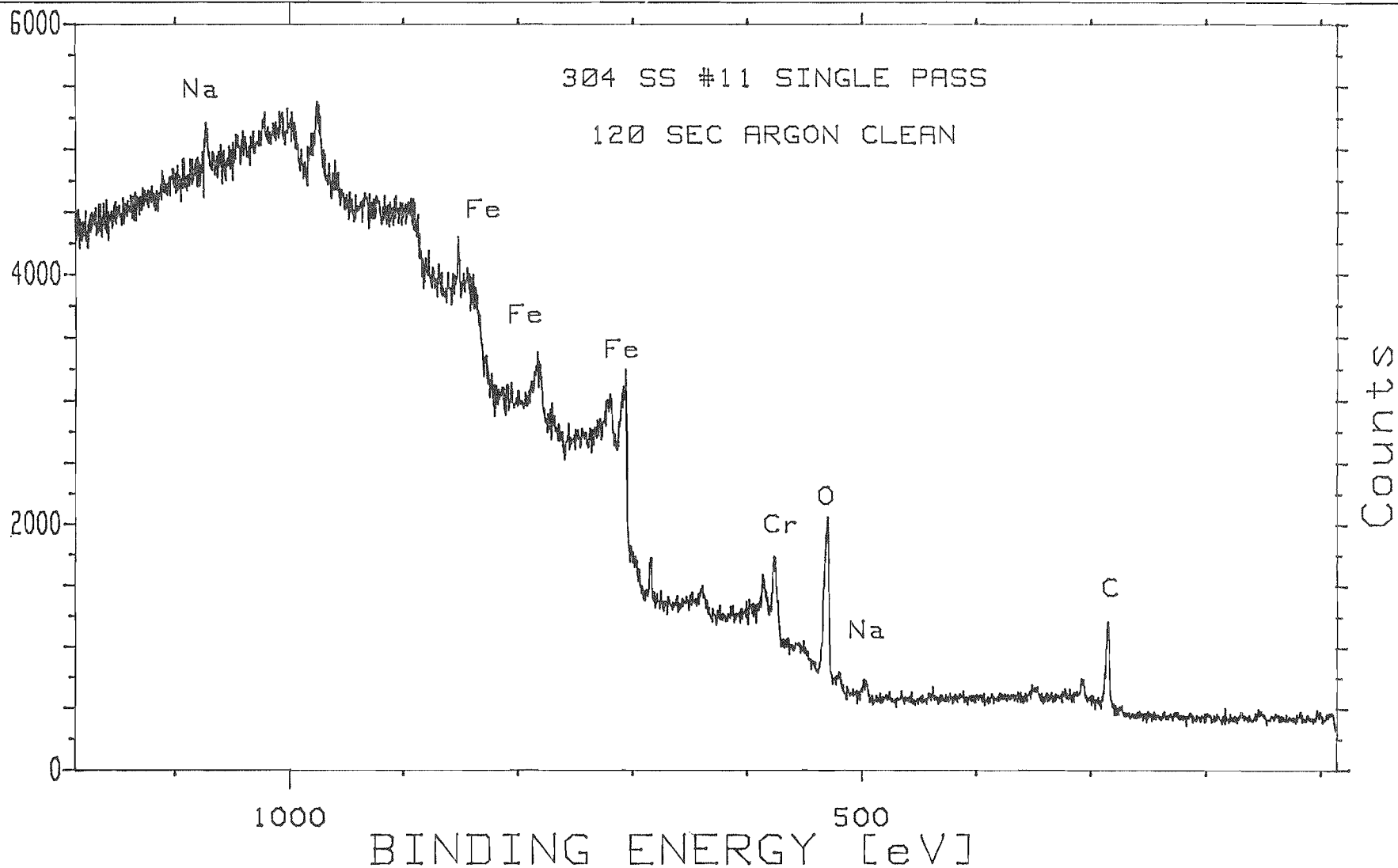


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Source Power 300 W Step .4

RESO= 1.0eV Al Anode

ARGON22_SP

16:15:18 6 Apr 1992

Fig. 5. 304SS Sample #11

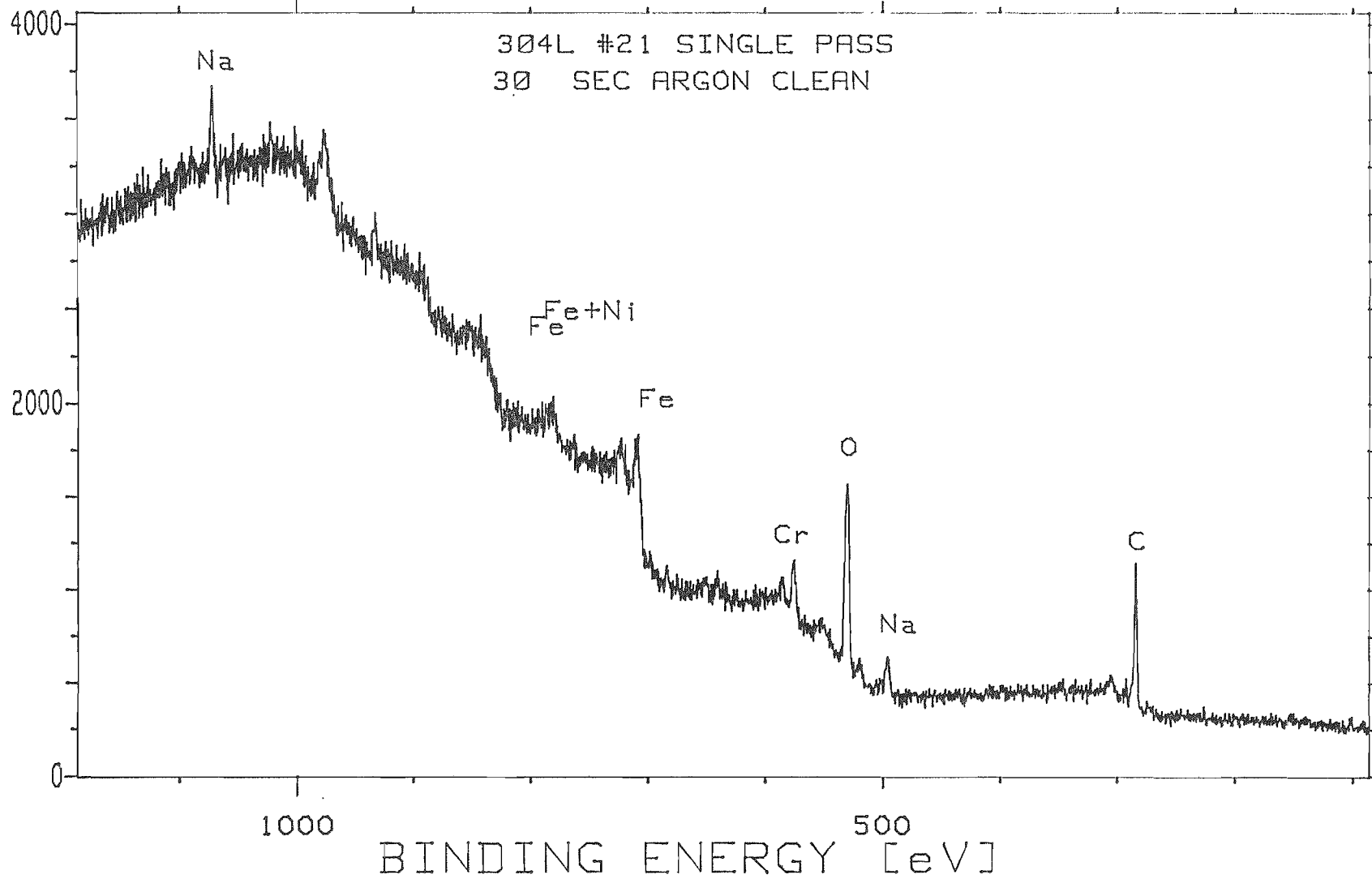


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Source Power 300 W

Step .4

ARGON12_SP

RESO= 1.0eV

Al Anode

13:22:59

6 Apr 1992

Fig. 6. 304L SS Sample #21

Fig. 7.

Elemental Atomic Percentages on Sample Surfaces-

Material	Cleaning Data	Fe	Ni	Cu	Cr	O	C	Ca	Na	Si
304 SS #14	Uncleaned					43.1	51.1		5.7	
304 SS #11	SCC Single Pass	15.3			13	49	19.1		3.6	
Run 1	SCC Single Pass 30 Arg. Cl.	34.5	5.2		16.2	30.9	10.3		2.9	
3500 psi	SCC Single Pass 120 Arg. Cl.	43.8	5.6		13.6	26	7.2	1.3	2.4	
304 SS #12	SCC Single Pass	18	1.2		10.5	46.6	20.2	0.1	3.4	
Run 2	SCC Single Pass 30 Arg. Cl.	33.6	4.5		14.2	31.3	10.6	1.1	4.7	
2000 psi	SCC Single Pass 120 Arg. Cl.	40.8	5.5		15.4	27.8	7.3	0.7	2.5	
304 SS #15	Chemical Clean	42.2	5.1		15.9	30	5.6		1.1	
	Chemical Clean 30 Arg. Cl.	48.6	6.5		14.5	29.4	1			
304L SS #24	Uncleaned					35.5	64.5			
	Uncleaned 120 Arg. Cl.					19.9	72.1		8	
304L SS #21	SCC Single Pass					57.1	31.9		11	
Run 1	SCC Single Pass 30 Arg. Cl.	35.2	4.7		11.6	31.6	12.1		4.8	
3500 psi	SCC Single Pass 120 Arg. Cl.	38.9	5.7		13.5	29.6	6.3	1.6	4.4	
304L SS #23	SCC Single Pass	12.8	1.6		9.9	52.4	19.2		4.1	
Run 2	SCC Single Pass 10 Arg. Cl.	34.5	4.7		9.1	36.2	12.3		3.2	
2000 psi	SCC Single Pass 30 Arg. Cl.	38.9	4.8		10.5	30.2	13.2		2.5	
	SCC Single Pass 120 Arg. Cl.	43.3	5.2		14.7	29.1	5.5		2.2	
304L SS #25	Chemical Clean	31.2	4.2		13.2	42.7	8	0.7		
	Chemical Clean 30 Arg. Cl.	50.6	6.4		13.2	28.6	1.1			
OFHC #5	Uncleaned			65.4		21.3	13.3			
OFHC #1	SCC Single Pass			49		30.6	12.9			7.5
Run 1	SCC Single Pass 30 Arg. Cl.			86.2		8.3	5.5			
3500 psi	SCC Single Pass 120 Arg. Cl.			91.6		4.3	4.1			
OFHC #3	SCC Single Pass			60.5		27.9	11.6			
Run 2	SCC Single Pass 30 Arg. Cl.			91.2		6	2.8			
2000 psi	SCC Single Pass 120 Arg. Cl.			94.4		3.8	1.9			
Material	Cleaning Data	Fe	Ni	Cu	Cr	O	C	Ca	Na	Si
OFHC #6	Chemical Clean			90.8		7	2.2			

Fig. 7.

	Chemical Clean 30 Arg. Cl.	97.2	2.4	0.4	
	Chemical Clean 120 Arg. Cl.	98.5	1.4	0.2	
Glidcop #34	Uncleaned	28.6	27.1	35.5	8.8
Glidcop #31	SCC Single Pass	48.4	30.5	17.6	3.5
Run 1	SCC Single Pass 30 Arg. Cl.	90.2	5.1	4.7	
3500 psi	SCC Single Pass 120 Arg. Cl.	95	2.7	2.3	
Glidcop #33	SCC Single Pass	50.8	30.4	13.3	5.5
Run 2	SCC Single Pass 30 Arg. Cl.	91.3	5.1	3.2	0.5
2000 psi	SCC Single Pass 120 Arg. Cl.	96.6	1.8	1.6	
Glidcop #35	Chemical Clean	72.7	21.1	6.2	
	Chemical Clean 30 Arg. Cl.	96.6	2.7	0.7	

Notes

Run 1 was supercritically cleaned for 1 hour at 45 deg. C at a pressure of 3500 psi with a flow rate of 1.4 cm/min.

Run 2 was supercritically cleaned for 1 hour at 45 deg. C at a pressure of 2000 psi with a flow rate of 1.6 cm/min.

Single pass designates CO₂ passes sample 1 time and is then exhausted to atmosphere.

Chemical cleaning consisted of ultrasonic cleaning for 15 min in each of trichlorethane, acetone and ethyl alcohol at room temperature.

30 Arg. Cl. denotes sample was Argon ion etched in ESCA chamber for 30 sec to remove airborne surface contamination from stored samples.